Journal of the European Ceramic Society 18 (1998) 1161–1169

PII: S0955-2219(98)00038-7
0955-2219/98/$—see front matter

Role of the Clogging Effect in the Slip Casting Process

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Abstract

Particle segregation phenomena occurring during consolidation of the green bodies lead to non-homogeneous microstructures and poor reliability of the ceramic components. The two driving forces responsible for particle segregation in the slip casting process are the gravity force and the capillary suction of the plaster moulds. Both segregation mechanisms depend on other factors such as particle density, particle size distribution, particle interaction forces, and solid loading. The relative importance of each segregation mechanism was investigated by using two commercial silicon carbide powders having different particle size distributions, as well as different colours. The effects of the interparticle forces, solid loading and the driving force of the slip casting process were studied. The results obtained showed that for moderately concentrated suspensions and a driving force given by the capillary suction of the plaster moulds, the clogging effect plays the main role in particle segregation, whilst sedimentation predominates for low driving forces. Inadequacy of the existing models to explain the clogging effect is discussed and a new model is proposed. © 1998 Elsevier Science Limited. All rights reserved

1 Introduction

The homogeneity of particle packing in the green compacts is essential for the sintering behaviour and the ultimate material properties.[1–5] It is commonly accepted that the properties of green compacts can be improved by using colloidal shaping techniques.[6,7] These processing methods enable to control and manipulate the forces between particles within a liquid, and can be used to fractionate heterogeneities, like agglomerates and inclusions, from powders that would other wise lead to strength-degradation flaw populations.[8]

The major factors known to control the rheological properties of the suspensions are the interparticle forces,[9,10] particle size distribution,[11,15] particle shape and density,[8–16] and solid volume fraction.[8–17] The same factors, as well as the driving force for the deposition process also determine the homogeneity and the particle packing density in the green compacts. Repulsive interparticle forces are normally used to prepare dense and homogeneous suspensions with good flow properties for the casting operations. The particle packing density can be greatly improved by mixing the proper volume fractions of different particle sizes having adequate mean size ratios to fit the Furnas model.[11] Following this principle, the relative green density of SiC slip cast bodies could be increased from about 60% (monomodal powder) to 74·5% by using a bimodal particle size distribution designed to maximise the particle packing.[13] Isometric and rounded particles have a higher ability to closely fill the space. Milewski[16] has shown that particle packing density dramatically decreases with increasing particle aspect ratio.

To take full advantages of the colloidal shaping methods the homogeneity achieved in the slurry state must be preserved during consolidation. That is, when different size particles are separated from one another, they cannot be arranged as idealised by Furnas[11] to produce their optimised particle packing density. Segregation phenomena can be prevented by using high solid loading and/or less stable suspensions[17] or, alternatively, by using an external pressure to increase the consolidation rate.[18] This makes the driving force of the casting process an important factor for achieving a high degree of homogeneity.

In the slip casting process, the separation of particles by size (or density) can occur through two different segregation mechanisms: (i) settling of the coarser (denser) particles; (ii) clogging the cake by the finer (lighter) ones. The first is determined by the gravity force and, for very dilute suspensions, is well described by the Stokes law:

$$v_p = \left[(\rho_p - \rho_1)\cdot d_p^2\cdot g)\right]/18\eta \quad (1)$$

where
v_p = velocity of a settling particle
ρ_p = density of bulk particle
ρ_1 = liquid density
d_p = particle diameter
g = acceleration due to gravity
η = viscosity of the liquid

The settling movement becomes gradually hindered as the volume fraction of solids increases. For example, Buscall *et al.*[19] and Velamakanni and Lange[8] have shown that mass segregation during pressure filtration could be avoided only when the volume fraction of the binary mixture that produces the optimum packing was $\geq 0{\cdot}50$.

The clogging of the cake by the finer particles is a very common but less well understood segregation phenomenon. It depends upon the interplay between the above referred factors influencing the rheology of the suspensions and the packing behaviour of the particles. Contrarily to sedimentation that occurs only in one direction (vertical), the clogging effect occurs in any direction parallel to liquid flow, in processes like slip casting and filtration. Fine particles have a lower inertia momentum and are more easily transported by the fluid. Thus, they will be the first to be deposited at the mould wall. Some can even penetrate and clog the larger pores within the mould. However, most of the literature often refers mass segregation as being due only to sedimentation.[8,20,21] Velamakanni and Lange[8] used well dispersed slurries of two alumina powders with mean particle sizes (D_{50}) of 0·3 and 1·3 μm separately and mixed in the proportion 40/60. In all the cases the packing density was improved when solid loading increased from 0·2 to 0·5 vol%, but to different extents. The increments in green density with solid loading were higher for the bimodal suspensions followed by that containing only the coarser powder, and were attributed to a diminution of mass segregation by sedimentation. However, the effect was expected to be more pronounced in the case of the suspension containing only coarse particles if the sedimentation was the dominant segregation mechanism. Kimura *et al.*[20] coagulated multicomponent slurries containing equal volume fractions of Al_2O_3 and ZrO_2 and a total of 7 vol% solids to avoid mass segregation when the dispersed slurries were poured onto an absorbent plaster plate. The authors refer that the layer in contact with the plaster mould was richer in coarser particles. These results suggest that the sedimentation mechanism would have played the main role in their experimental conditions. This is not surprising since the solid loading used was very low. Mizuta *et al.*[21] concluded that a narrow particle size distribution favoured dense and uniform particle packing to be obtained, compared with broader particle size distributions. However, this conclusion cannot be generalised. In fact, suspensions within the solid loading range used (5 to 30 vol%) are prone to mass segregation by the two discussed mechanisms. Furthermore, it was not clear if the coarse particles were individualised particles or hard agglomerates with a lower bulk density.

The existing models[22–24] aiming to explain the mass segregation within the suspensions during consolidation are scarce and unable to describe adequately the clogging effect. Kaneko[22] used multicomponent slurries of Al_2O_3 (D_{50} = 0·3–0·5 μm) and ZrO_2 (D_{50} = 0·8–1·2 μm) and solid contents varying between 20 and 40 vol% to process sheets by tape casting and concluded that low solid loading and slow drying conditions favoured mass segregation. The simple model proposed by Kaneko[22] assumes that coarse particles sediment first, forming a skeleton, followed by the deposition of the finer particles within the interstices left by the former ones. This model seems suitable to describe mass segregation by sedimentation, but cannot be adopted to describe the clogging effect. Hampton *et al.*[23,24] developed a mathematical model to predict the rate of thickness growth of a clogged cake, schematically shown in Fig. 1. They consider that the fine particles were transported by the filtrate through an inner cake region, where the proportion of coarse and fine particles was about the same as in the slip, and deposited in the voids

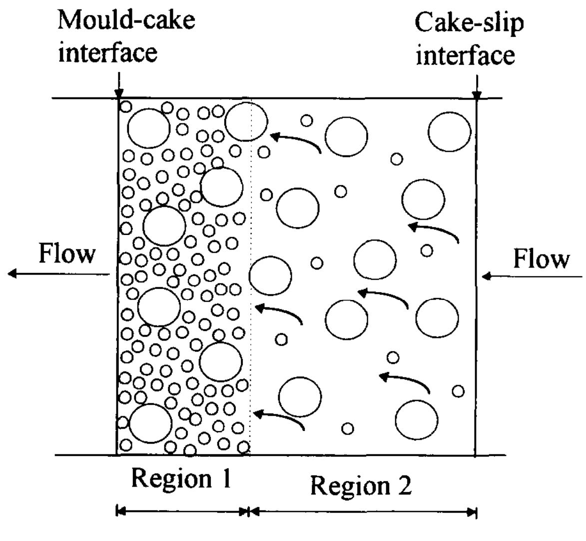

Fig. 1. Schematics of the model proposed by Hampton *et al.*[23,24] to explain the clogging effect.

near the bottom of the cake. However, this model fails when all the slip inside the mould is going to be consumed, like in solid casting. Ferreira[25] found that the clogging of the cake was the main mass segregation mechanism occurring during slip casting of bimodal and moderately concentrated (62·5 wt%) SiC slurries. Besides the transport of the finer particles by the liquid flow, he also suggested the possibility of the coarse and isometric particles to migrate in the opposite direction. This inwards movement of coarse particles is less probable with the anisometric whiskers used by Sacks *et al.*[26] They observed that the average pore size of green compacts (90% Al_2O_3, 10% SiC whiskers) was independent of the solid loading in the range between 18 and 54 vol%, whilst the pore size distribution was only slightly dependent upon this experimental variable.

The purpose of the present work was to use a bimodal particle size distribution, designed to maximise the particle packing, to study the influence of solid loading, the amount of dispersant and the driving force of the deposition process on the clogging effect.

2 Experimental Procedure

2.1 Slurries preparation and characterisation

The starting raw materials were two commercial silicon carbide powders, one of the green variety, NF0 ($D_{50}=0{\cdot}7\,\mu$m) and the other of the dark grey variety, 1200P ($D_{50}=15\,\mu$m), (Elektroschmelzwerk, Kempten, GmbH, Germany). Particle size distributions of the powders were determined using a Mastersizer (Malvern Instruments Ltd, UK) in the presence of 0·25 wt% dispersant. The size and shape of particles were also analysed by scanning electron microscopy (SEM), (Model S4100-1, Hitachi, Ltd., Tokyo, Japan).

The fine and coarse powders were combined in the proportion 45/55 that gave the highest packing density.[13] Suspensions containing from 62·5 to 80 wt% solids were prepared by adding the powders to distilled water or to aqueous solutions containing varying amounts (from 0·0 to 0·5 wt%) of dispersant, Targon 1128 (BK Lademburg, Germany), a product based upon ammonium polycarbonates of low molecular weight. The mixtures were hand stirred, followed by simultaneous stirring and ultrasonication for 20 min.

2.2 Preparation and characterisation of the green bodies

A first part of this study was focused on studying the effects of the solid loading and the amount of deflocculant on relative density and extent of the segregation. To perform these evaluations, rod-like green bodies were prepared by pouring the suspensions into plaster moulds, with a plaster/water ratio of 1·25, to form solid cylindrical samples (120 mm long, $\phi = 8$ *mm*). The evolution of the interface cake/suspension was detected manually by introducing a fine wire trough the central axis of the cylinder being formed. The demoulding operation was performed as soon as the casting was considered complete, that is, when instead of the fluid suspension, the typical resistance of the consolidated layer was encountered. Just after demoulding, samples were weighed and placed in a stove at 120°C for 24 h. After complete drying, samples were cooled in a desiccator to avoid water uptake from the atmosphere and then reweighed. The density of the slip cast samples was measured by the Hg immersion method using the two end-cuts of each sample. Portions of the inner and the outer parts of the cylindrical rods were redispersed for particle size analysis.

A second part of this work aimed to evaluate the influence of the driving force on the extent of the segregation phenomena during the deposition process for slips containing 0·15 wt% dispersant and 62·5 wt% solids. Driving forces lower than the suction pressure exerted by the plaster moulds ($\approx$ 0·15 MPa)[25] were generated by vacuum filtration using either a glass sintered filtering crucible, G4, (mean pore size 10–16 μm) or a Buckner funnel. Driving forces higher than the suction pressure of the plaster moulds were generated with compressed air in pressure slip casting. A pressure of about 0·2 MPa was applied over the slip.

The filtering surfaces of the filtering crucible and Buckner funnel were covered with filter paper (Whatman 42, USA) having a mean pore size $\approx$2·5 μm. Portions of 100 g ($\approx$ 57 cm^3) of a 62·5 wt% solid loaded suspension were used in each test. Cakes were dried at 120°C for 24 h. Subsequently, powder samples were taken at different cake levels, from the bottom to the top, and redispersed for particle size analysis.

3 Results and Discussion

3.1 Characterisation of the powders

The particles' size and morphology of the fine and the coarse powders are shown in the micrographs of Fig. 2(a) and (b), respectively. The particles of the finer powder tends to form aggregates, whilst those of the coarser one are individualised. These results are not surprising, since the gravitational force predominates over the surface forces in the case of large particles, whereas an opposite

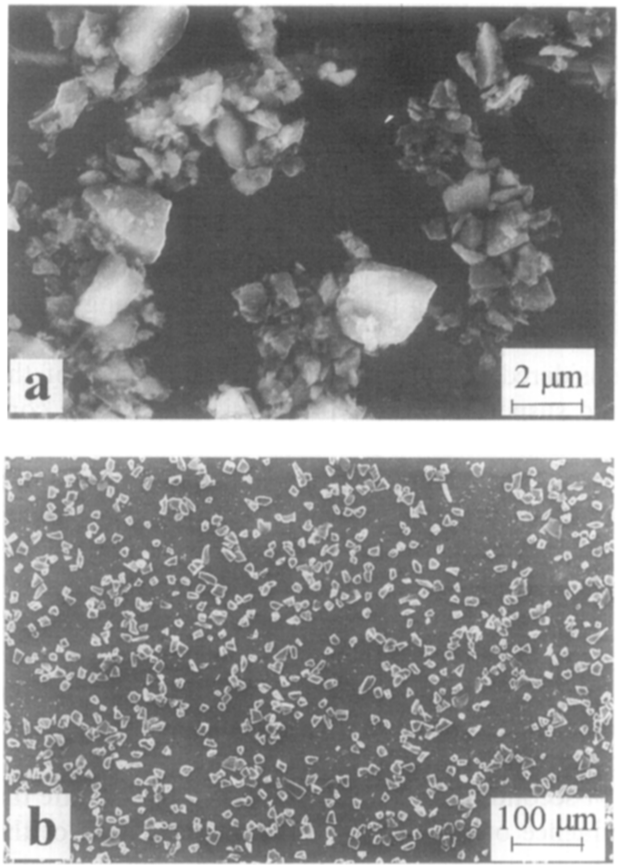

Fig. 2. SEM Micrographs of the powders used: (a) NF0, (b) 1200P.

situation occurs with small particles due to the predominance of interface (attractive) forces. Particle size distribution curves are reported in Fig. 9, which show that the average particle sizes of fine and coarse powders are 0·7 and 15 μm, respectively.

3.2 Effects of the amount of dispersant and solid loading

The evolution of the relative density and moisture content (after demoulding) of slip cast samples with increasing concentrations of Targon 1128 for different solid loading is reported in Fig. 3(a) and (b). For a given amount of dispersant, the packing density increases with increasing solid loading reaching maximum values in the range 70–72·5 wt% solids. Beyond this solid content the packing density tends to decrease again. The moisture content of the slip cast bodies shows an inverse trend. The highest packing density, 75·5% of the theoretical density (TD) is observed at 70 wt% solids and 0·1–0·15 wt% dispersant. These results are in agreement with the trends observed by Pivinskii and Bevz,[27] Rutman *et al.*,[28] and Tari et al[14] for bodies prepared from electrostatically stabilised suspensions.

For the green bodies prepared from well dispersed suspensions containing less than 70 wt% solids, the cylindrical cross-section presented two different regions, schematically shown in Fig. 4. Owing to the different colours of the starting powders, they could be easily distinguished with a naked eye. The inner region was richer in coarse grey particles while the outer one was constituted mainly by the green fine particles. These diameters were measured, with a naked eye, at distances from the extremities of the rods that varied usually between 15 and 20 mm. Figure 5 shows the evolution of the inner diameter measured near the top and near the bottom parts of cylindrical rods with increasing solid loading and amounts of dispersant. It can be observed that the bottom diameter of the segregated zone is always higher than the top diameter for a given set of experimental conditions. This difference corresponds to the settling component for particle segregation. However, it could be seen that the bottom part of rods ended by a layer rich in fine particles with a similar thickness as that observed in the near cross sectional fracture. This means that the clogging effect played a dominant role in the overall particle segregation phenomena in the solid loading range tested.

These results can be explained as follows. At the lower solid contents, full stabilised suspensions enable particles to flow and pack as individuals. The slips are too fluid and strong electrostatic repulsions exist between the particles in suspension[18,25] and between them and those in the consolidated wall. The number of particles that reach

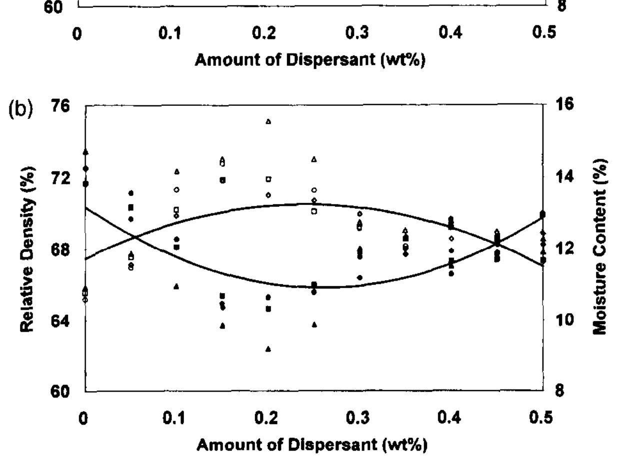

Fig. 3. Effects of the amount of dispersant and solid loading on green packing density (open symbols), and moisture content (full symbols). Key: (a) 62·5 wt% (△▲), 65 wt% (○●), 67·5 wt% (□■), 70 wt% (◇◆); (b) 72·5 wt% (△▲), 75 wt% (○●), 775 wt% (□■), 80wt% (◇◆).

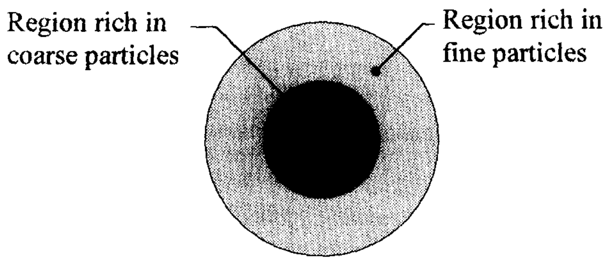

Fig. 4. Schematic of the cylindrical cross-section of a rod sample presenting two distinct segregated regions.

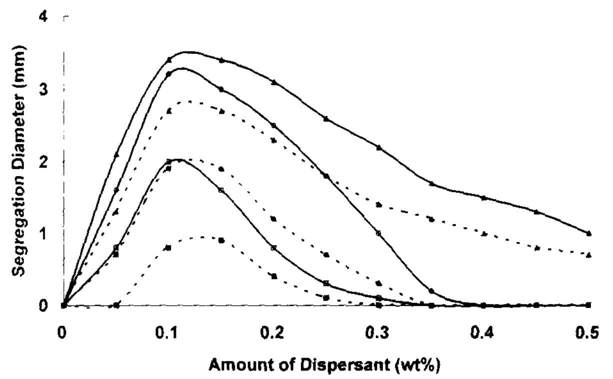

Fig. 5. Evolution of the diameters of the inner segregated region with increasing amounts of dispersant and solid loading, measured near the top (dashed lines) and near the bottom (continuous lines) of the cylindrical slip cast rods. Key: 62·5 wt% (△▲), 65 wt% (○●), 67·5 wt% (□■).

the interface between the slip and the consolidated layer, per unit time, is relatively small and more time is allowed for each particle to be deposited. All these interrelated factors enable the formation of close packed and low permeable green microstructures. These conditions are prone to mass segregation, as will be explained in the following.

The ratio between the mean equivalent spherical diameters of the powders NF0 and 1200 P is about 20. Assuming that particles are spherical with two different and uniform radii, R_f (fine) and R_c (coarse), and a size ratio, $R_c/R_f = R_{cf} = 20$, the mass ratio will be $W_{cf} = 8000$. So, a coarse particle is much heavier than a fine particle. When the slip is poured into the mould, finer particles will be the first to be pushed against the porous wall because of their lower inertia. If the resultant of the interaction forces is repulsive and the rate of deposition is relatively low, they can form closed packed domains,[12] since these conditions are favourable to particle rearrangements. The formation of closed packed arrays imply that the particles of a second layer will be deposited within the triangular pores of the first layer. In doing so, a fine particle immerses a significant fraction of its radius (0·367 R_f) in the top plan defined by the first layer, parallel to the mould wall, as was shown previously.[25] Contrarily, for the coarse particles, the first layer formed appears too smooth because only a very small fraction of its own radius (0·0016 R_c) can be immersed in that plan. So, when a coarse particle reaches the interface cake/suspension it is not laterally involved and its immediate fixation at the wall is uncertain. The more movable fine particles might pass underneath and push it inwards enhancing the segregation by the clogging mechanism, as suggested in Fig. 6. Besides the transport of the finer particles by the liquid flow, this model also suggests the possibility of the coarse particles to migrate in the opposite direction, with the consolidated layer acting like a sieve.

The micrographs presented in Fig. 7 show some aspects of the cross sections of cylindrical rods prepared from suspension containing a fixed amount of dispersant (0·15 wt%) and solid contents of (a) and (b) 65 wt%, (c)70 wt%, and (d) 80 wt%. Pictures (a) and (b) were taken in the central and peripheral zones, respectively. The inner region is almost depleted of fine particles, whilst the outer region is formed by both types of particles and seems very compacted. Picture (c) reveals that the measurements of inner diameters (Fig. 5) made without the aid of any optical instrument were not very accurate. In fact, the diameter found with naked eye was nil at 70 wt% solids, while the true diameter is about 200 μm. Comparison between Fig. 2(b) and Fig. 7(a) and (c) suggests that the average size of particles retained in the inner region have an average size higher than that of the coarse powder. This is confirmed by the particle size distributions of the redispersed inner and outer parts of the cylindrical rods, presented in Fig. 8. For comparison, the particle size distributions of fine and coarse powders are also shown. It can be seen that both inner

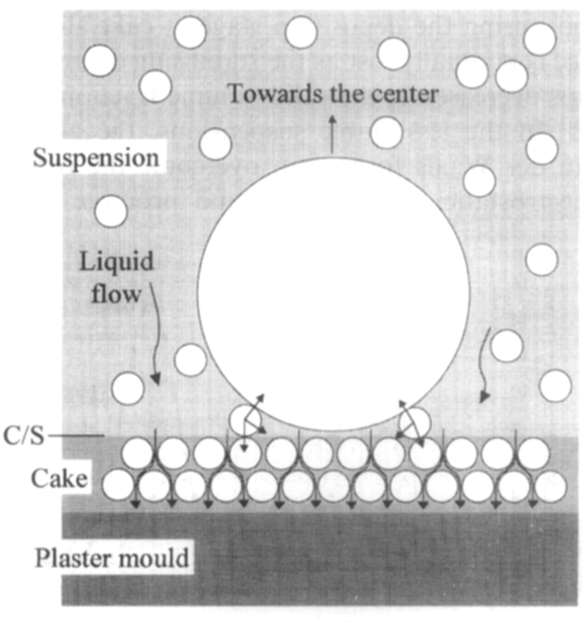

Fig. 6. The clogging mechanism proposed. Fine particles might pass underneath a coarser one at the interface cake/slip and push it inwards, with the consolidated layer acting like a sieve.

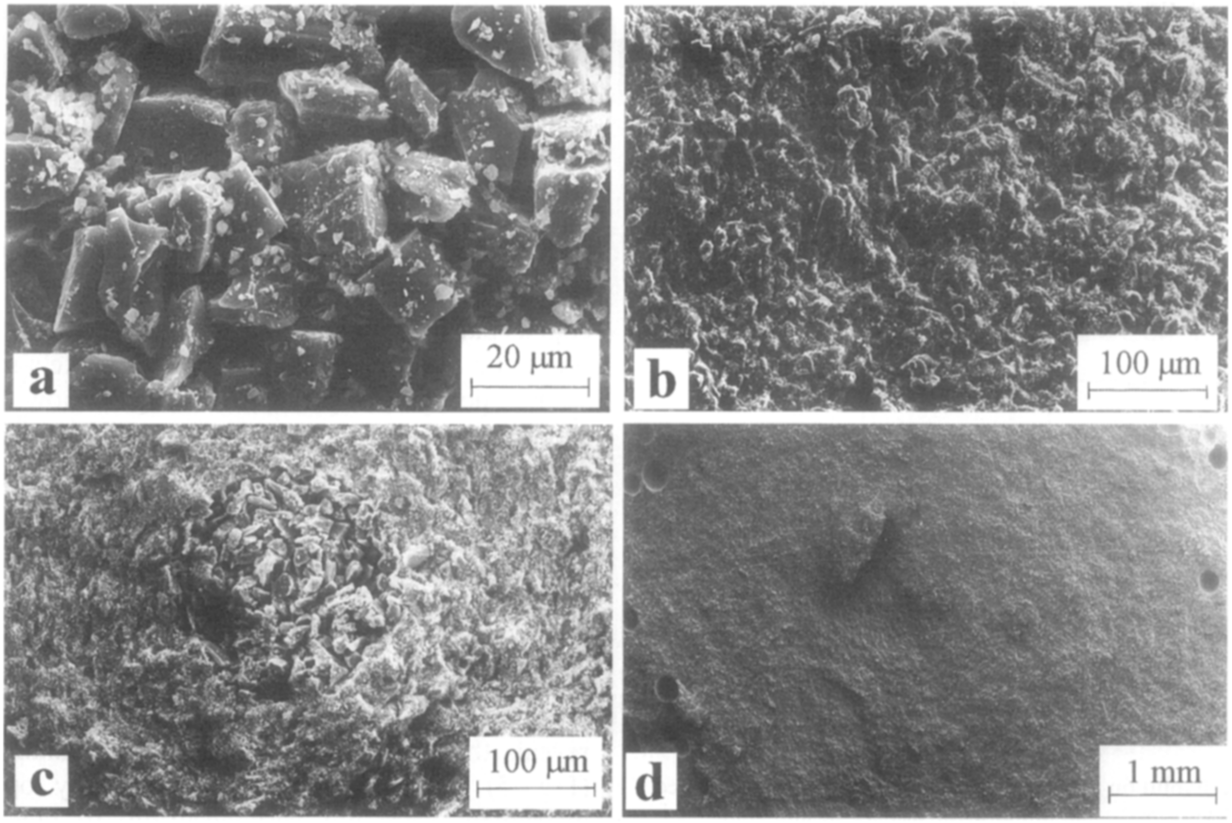

Fig. 7. The micrographs showing some aspects of the cross sections of cylindrical rods prepared from suspension containing 0·15 wt% dispersant and solid contents of (a) and (b) 65 wt%, (c) 70 wt%, and (d) 80 wt%. Pictures (a) and (b) were taken in the central and peripheral zones, respectively.

and outer parts are composed of bimodal particle size distributions, but the coarse fraction is significantly smaller in the outer region. Furthermore, the coarse fractions are centred at about 10 and 20 μm in the cases of the outer and the inner regions, respectively, supporting the clogging model proposed above.

The 'freedom' of particles within the suspension and during the deposition stage at cake/slip interface is gradually lost as the stabilising degree of the slips decreases or the solid volume fraction increases. In the less stable suspensions, the attractive van der Waals forces can overcome the repulsive (electrostatic, steric) forces and promote particle

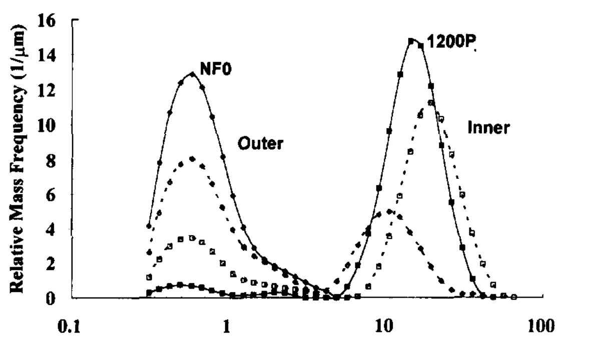

Fig. 8. Comparison between the particle size distributions of the starting powders and those of the redispersed outer and inner parts of the cylindrical rods obtained by slip casting.

agglomeration leading to the formation of more permeable deposits. As a consequence, the green density, as well as the extent of the segregation phenomena will be lower, as observed. High 'freedom' favours, at the same time, particle rearrangement and mass segregation. For a given set of experimental conditions, the packing density will depend upon the balance between these two opposing tendencies. It seems that the best compromise in terms of green density is obtained from the well-stabilised slips containing 0·10–0·15 wt% dispersant and 70 wt% solids. However, the highest packing density seems not to be the best criteria in selecting the optimal processing conditions, since mass segregation by the clogging mechanism still occurs at this solid loading, as shown in Fig. 7(c).

At high solid loading, suspensions become too crowded. Particles have to approach each other, overlapping their electrical double layers. This closer approach of particles in suspension does not necessarily give a more ordered particle packing, since with the increasing deposition rates the frequency of particle collisions also increases. The searching of one particle for a favourable packing site at the interface cake/suspension is hindered by the large number of new incoming particles. In these conditions, the time allowed for particle rearrangements is not enough. At the same time, the stabilisation of concentrated suspensions becomes increasing difficult.[8,9,25] Furthermore, in these conditions entrapped air bubbles are more difficult to escape. This is confirmed by the micrograph presented in Fig. 7(d) which shows an overall view of the sample prepared with 80 wt% solids. Consequently, a lower packing degree will be expected. Since neither particle segregation nor entrapped air bubbles are desirable, the results presented in this work suggest that high solid loading and the integration of a de-airing step would be considered in an optimal processing procedure.

3.2.1 Effect of the driving force for consolidation

In absence of any driving force other than the gravity force, only sedimentation takes place. The first attempt to use a driving force lower than the suction pressure of the plaster moulds made with the porous glass sintered crucible, showed that the pressure drop along the filter, due to the movement of the air, was about 650 mmHg. So, the driving force available for consolidation was too low. After complete drying, the cake was analysed. It could be observed that a thin layer of fine particles was detached together with the filter paper. This sample was labelled as 'AFP' (adjacent to filter paper). Other powder samples were taken at bottom 'Bottom', intermediate level 'Middle', and at the top 'Top' of the cake. The particle size distributions of the these samples are presented in Fig. 9. These results show that the suction pressure exerted by the filter paper provided enough driving force to promote segregation by the clogging mechanism. However, after complete saturation of the filter paper, the remaining driving force available was too low and sedimentation dominates the mass segregation.

A driving force near to 1 atmosphere could be generated by using a Buckner funnel. Figure 10 shows a scheme of the apparatus used and of the cake obtained. The higher driving force available enabled a first layer rich in fine particles to be formed by the clogging mechanism. However, the pressure drop along the cake is proportional to its thickness. The clogging mechanism gradually slows down whilst the sedimentation becomes more and more important. At a given thickness, sedimentation completely dominates the mass segregation process. The overall result is something like a sandwich with a layer rich in coarse particles embedded in two layers of fine particles. The particle size distributions of the powder samples taken at bottom, top, and intermediate level are presented in Fig. 11. It can be concluded that both samples gathered at Bottom and Top parts of the cake are mainly composed by the finer component powder, whilst the sample collected in the Middle region approaches the coarser component powder size distribution.

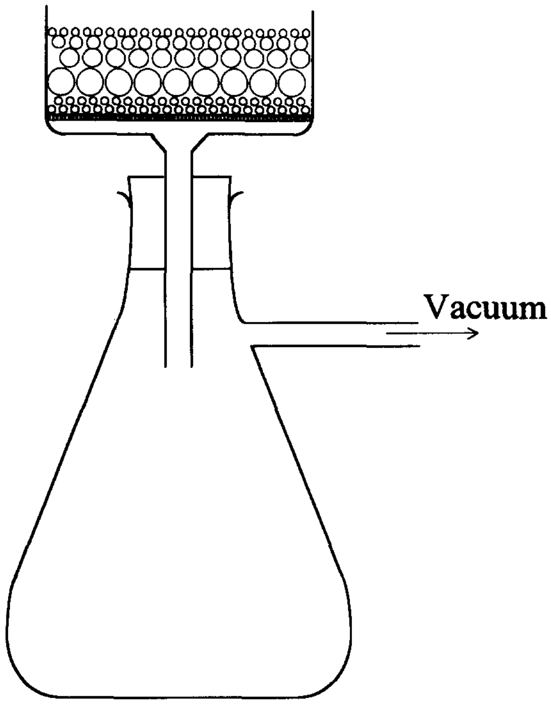

Fig. 10. Schematics of the filtering system involving the Buckner funnel. In these conditions a sandwich type cake was formed with a layer rich in coarse particles embedded in two layers of fine particles.

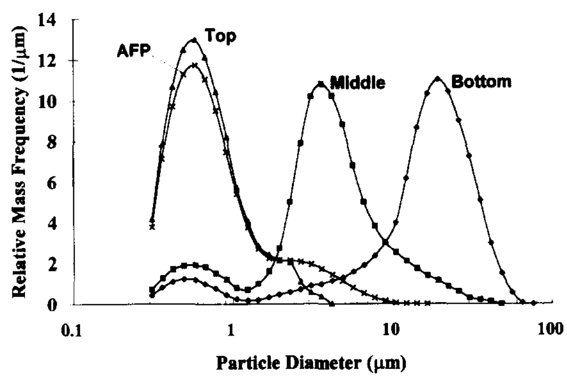

Fig. 9. Particle size distributions of the samples taken from the vacuum filtration cake, obtained with the porous glass sintered crucible (G4), at different levels, (AFP = layer adjacent to filter paper).

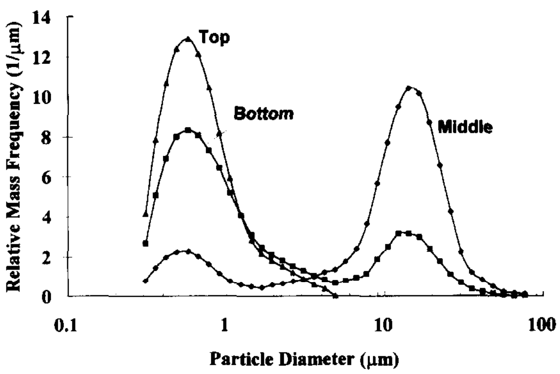

Fig. 11. Particle size distributions of the samples taken from the vacuum filtration cake, obtained with the Buckner funnel at different levels.

When a pressure of about 0·2 MPa was applied over the slip in contact with the plaster mould (total effective pressure of about $\approx$ 0·35 MPa) no mass segregation was observed within the cake.

4 Summary and Conclusions

These results presented in this work show that the packing behaviour of a bimodal particle size distribution depends on the interplay between the amount of deflocculant, solid loading and the available driving force for consolidation. At a given solid loading, the packing density obtained by slip casting into plaster moulds increases with increasing amounts of dispersant added until a maximum is reached, decreasing for further additions. For well dispersed and low solid loaded slurries, the high fluidity and the low deposition rates promote mass segregation by the sedimentation and clogging mechanisms and particles can not pack in a homogeneous way. With the solid loading increasing, suspensions become more crowded and the deposition rate increases. Collisions and mutual interference among particles during the deposition stage reduce both the potentials for particle rearrangement and, the tendency for particle segregation. The packing ability is determined by these two opposing tendencies. At high solid loading the slip viscosity is enough high to hinder particle rearrangement, particle segregation, and the escape of the entrapped air bubbles. Consequently, a lower packing degree should be expected. Furthermore, a de-airing step would be considered in an optimal slip preparation procedure.

The driving force for consolidation also plays an important role in mass segregation. At moderate solid loading, bimodal and well-dispersed slurries segregate preferentially by sedimentation at low driving forces, and by the clogging mechanism when the effective pressure is around 1–2 atmospheres. Segregation phenomena can be avoided by increasing the deposition rate. No mass segregation was observed when the effective pressure was increased to $\approx$ 0·35 MPa.

Besides the transport of the finer particles by the liquid flow, a model to describe the clogging effect should also consider the possibility of the coarse particles to migrate in the opposite direction, with the consolidated layer acting like a sieve.

Acknowledgements

This work was supported by the European Community, contract CHRX-CT94-0574 in the frame of the Human Capital and Mobility Programme.

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